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NO. 3899 P. 6/24

Application No. 10/069,087
After Final Office Action of July 17, 2006

Docket No.: 05587-00327-US

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REMARKS

Applicant respectfully requests reconsideration in view of the following remarks. Claims 1-3, 11, 12 and 14-19 and 21-26 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. Claims 1-3, 11, 12 and 14-19 and 21-26 are provisionally rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1-31 of copending Application No. 10/381,502. Claims 1-3, 11, 12 and 14-19 and 21-26 are rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1-20 of U.S. Patent No. 6,306,940 to Disch et al. Claims 1-3, 11, 12, 14-19 and 21-26 are rejected under 35 U.S.C. 103(a) as being unpatentable over Auerbach et al. U.S. Patent No. 4,666,995 ("Auerbach") taken with Paul et al. U.S. Patent No. 4,727,106 ("Paul") in view of Chapman et al. U.S. Patent No. 3,656,982 ("Chapman"), all in view of Mück et al. U.S. Patent No. 5,994,455 ("Mück").

Comments to the Interview

The applicant appreciates that Examiner Nutter permitted the applicant to interview the application on October 19, 2006. The applicant discussed submitting the enclosed copy of a Standard used by General Motors which refers to VDA 275. As discussed in the interview, this standard establishes that the VDA 275 to be known and recognized in the automotive industry. The applicant has discussed submitting an executed terminal disclaimer to obviate the double patent rejection over Application No. 10/381,502 and U.S. Patent No. 6,306,940. The applicant also discussed the possibility of amending claim 1 by incorporating the percentage features of claim 2 in order to overcome the double patenting over U.S. Patent No. 6,306,940 instead of

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filings a terminal disclaimer. The prior art was also discussed. The applicant discussed that Chapman describes only some pearlescent pigments for cosmetic usage. The applicant discussed that Chapman was not related to the claimed invention.

U.S.C. 112, Second Paragraph Rejection

Claims 1-3, 11, 12 and 14-19 and 21-26 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. The applicant believes that German Automotive Industry Recommendation No. 275 (VDA 275) is a well recognized standard for one of ordinary skill in the art. Enclosed is a copy of a Standard used by General Motors which refers to VDA 275. As discussed in the interview, this standard establishes that the VDA 275 to be known and recognized in the automotive industry. For the above reasons, this rejection should be withdrawn.

Double Patenting Rejections

Claims 1-3, 11, 12 and 14-19 and 21-26 are provisionally rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1-31 of copending Application No. 10/381,502. Claims 1-3, 11, 12 and 14-19 and 21-26 are rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1-20 of U.S. Patent No. 6,306,940 to Disch et al.

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The applicant has enclosed an executed terminal disclaimer which obviates the double patent rejection over Application No. 10/381,502 and U.S. Patent No. 6,306,940. For the above reasons, these rejections should be withdrawn.

35 U.S.C. 103(a) Rejection

Claims 1-3, 11, 12, 14-19 and 21-26 are rejected under 35 U.S.C. 103(a) as being unpatentable over Auerbach taken with Paul in view of Chapman, all in view of Mick.

Although, the use of POM as a carrier material for pigments is known, the existing shortcoming of chemical instability and subsequent formaldehyde evolution during processing and from molding has not been satisfactorily eliminated while at the same time retaining the required property profile.

The object of the applicant's claimed invention was to develop colored POM molding compositions which contain a colorant and in which the formaldehyde emission observed hitherto has been substantially reduced, in fact, the formaldehyde emission, determined on test specimens in accordance with the German Automotive Industry Recommendation No. 275 (VDA 275), is not more than 20 mg/kg, without impairing the known advantageous properties of POM. (see the specification at page 3, lines 22-25). The prior art references the Examiner has applied against the claimed invention does not recognize nor solve the problem.

The Examiner has stated that Auerbach teaches the use of a colorant. As the Examiner correctly cited Auerbach at the paragraph bridging col. 8 and 9 which states,

It is within the ambit of the present invention that the oxymethylene polymer molding composition also include, if desired,
1) plasticizers,
2) other formaldehyde scavengers,
3) mold lubricants,
4) antioxidants,

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- 5) fillers,
- 6) colorants,
- 7) reinforcing agents,
- 8) light stabilizers,
- 9) pigments,
- 10) other stabilizers,
- 11) and the like, so long as such additives do not materially affect the desired properties of the resulting molding composition and the articles molded therefrom. The additional additives can be admixed at any convenient stage in the molding composition preparation, but usually are added when the oxymethylene polymer is being blended or admixed with the polyamide-carrier resin dispersion.¹

It is noted that there are 11 optional ingredients cited by Auerbach. Auerbach gives no motivation to particularly select any of the optional ingredients.

As the Examiner pointed out, Paul discloses at col. 11, lines 3-21,

The stabilized oxymethylene polymer compositions also include if desired,

- 1) plasticizers,
- 2) pigments,
- 3) lubricants and
- 4) other stabilizers, e.g.,
- 5) stabilizers against degradation by ultraviolet light,
- 6) e.g., 2,2'-dihydroxy-4,4'-dimethoxy -benzophenone;
- 7) 2-hydroxy-4-methoxy-benzophenone;
- 8) 2-hydroxy-4-methoxyl-chlorobenzophenone,
- 9) nucleants,
- 10) UV screens and
- 11) absorbers,
- 12) metal soaps,
- 13) reinforcers and
- 14) filler such as
- 15) glass,
- 16) talc,
- 17) white mica and
- 18) gold mica,
- 19) polymeric substances such as
- 20) ethylene vinyl acetate,

¹ The numbers have been inserted by the applicant.

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- 21) polyurethanes,
- 22) impact modifiers, and
- 23) color pigments which are compatible with oxymethylene polymers,
e.g.,
- 24) red pigments such as
- 25) azo dye and
- 26) cadmium sulfide-cadmium selenide reds and
- 27) "Mercadium" reds,
- 28) blue pigments such as
- 29) phthalocyanine blues,
- 30) green pigments such as
- 31) chromium oxide greens,
- 32) white pigments such as
- 33) titanium dioxide whites, and
- 34) black pigments such as
- 35) carbon blacks which can be incorporated in amounts of up to about 5% by weight, based upon the total weight of the composition.²

It is noted that there are 35 optional ingredients cited by Paul. Paul gives no motivation to particularly select any of the optional ingredients.

Paul further discloses at col. 3, lines 28-36,

The term oxymethylene polymer as used herein is intended to include any oxymethylene polymer having --CH₂O-- groups comprising at least about 50 percent of the recurring units, for example, homopolymer, copolymers, terpolymers and the like.

Chapman describes only some pearlescent pigments for cosmetically usage (e.g. abstract). Under the heading of Description Of the Preferred Embodiments, Chapman states: "The present invention is particularly useful and beneficial in conjunction with pearlescent pigments which are to be incorporated in compressed cosmetic powders." (emphasis added)

² The numbers have been inserted by the applicant.

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There are no compositions described in Chapman which encompasses oxymethylene/oxyethylene copolymers. In addition, there is no indication given which kind of oxymethylene/oxyethylene copolymers has to be used for the reduction of the formaldehyde emission raised through the mixture of said copolymer with a colorant (pigment). The applicant does not believe that Chapman is related to the applicant's claimed invention or for that matter is combinable with the other references applied against the claims. Chapman is in a non-analogous art.

The Examiner states at the second full paragraph of page 6 of the Office Action that he relies upon Mück solely to show the trifluoromethanesulfonic acid initiator. However, Mück does not disclose that the formaldehyde emission, determined on test specimens in accordance with the German Automotive Industry Recommendation No. 275 (VDA 275), is not more than 20 mg/kg (see independent claim 1).

The Examiner argues at the bottom of page 6 of the Office Action that the low level of formaldehyde emission is inherent. However, the applicant respectfully disagrees. In a second step, a colorant is added to these polyoxymethylenes to form a colored composition with a low formaldehyde emission level. However, it is important to understand that the addition of a colorant usually leads to an increased destruction of the polyoxymethylene and following to an increased emission of formaldehyde. In the state of the art, the increased formaldehyde emission is reduced by addition of N-containing compounds (see for example Kosinski). In view of the present invention it was unexpectedly found that such an increase of emitted formaldehyde occurred by adding a colorant can be avoided respectively reduced if the specific prepared polyoxymethylenes are used. So the argument of the Examiner is to simple that only a

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polyoxymethylene with a low emission level of formaldehyde is used to prepare a colored polyoxymethylene composition which shows also a low formaldehyde emission. In contrast thereto it was not obvious that the colored, specific prepared polyoxymethylenes shows a reasonable lower increase of formaldehyde emission compared with other polyoxymethylenes prepared with other methods after coloration.

The oxymethylene/oxyethylene copolymers can be prepared with several alternative initiators (e.g. with Lewis acids, see Mück column 1 lines 35-39). There is no evidence in Mück that copolymers containing oxymethylene and oxyethylene units and a colorant (pigment) leads to an increased formaldehyde emission and that this emission can be reduced by mixing specific prepared copolymers (with a strong protonic acid) with the colorant (pigment). For example, the preparation of the copolymers with Lewis acids (described in Mück) leads to a higher formaldehyde emission (see e.g., the present application, in particular the comparative examples, wherein the copolymer is prepared with BF_3 (page 8 line 1, results page 10 table 1)).

In the state of the art the use of N-containing stabilizers are known to increase e.g. light or melt stability (e.g. Kosinski, see page 6, line 4-19). Surprisingly, oxymethylene-oxyethylene copolymers prepared with a specific method (with strong protonic acids as initiator) leads to a low formaldehyde emission level if a colorant is added. There is NO indication in Auerbach, Paul, Mück, nor Chapman that this specific combination (oxymethylene-oxyethylene copolymers in accordance with claim 1 and a colorant) to get colored copolymers results in a low emission level of formaldehyde, in particular, formaldehyde emission, determined on test specimens in accordance with the German Automotive Industry Recommendation No. 275 (VDA 275), is not more than 20 mg/kg.

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Further a person of ordinary skill in the art couldn't find any evidence in to prepare copolymers containing oxymethylene and oxyethylene units mixed with a colorant (pigment), wherein the copolymer is prepared with a strong protonic acid to reduce the formaldehyde emission of the resulting colored copolymer compound.

In summary, there are 11 optional ingredients cited by Auerbach. Auerbach gives no motivation to particularly select any of the optional ingredients. There are 35 optional ingredients cited by Paul. Paul gives no motivation to particularly select any of the optional ingredients. Chapman is related to a totally different field of invention that the applicant does not believe one of ordinary skill in the POM art would even look at Chapman. Again, Chapman describes only some pearlescent pigments for cosmetically usage. Mück does not disclose that the formaldehyde emission, determined on test specimens in accordance with the German Automotive Industry Recommendation No. 275 (VDA 275), is not more than 20 mg/kg.

The Examiner must consider the references as a whole, In re Yates, 211 USPQ 1149 (CCPA 1981). The Examiner cannot selectively pick and choose from the disclosed multitude of parameters without any direction as to the particular one selection of the reference without proper motivation. The mere fact that the prior art may be modified to reflect features of the claimed invention does not make modification, and hence claimed invention, obvious unless the prior art suggested the desirability of such modification (In re Gordon, 733 F.2d 900, 902, 221 USPQ 1125, 1127 (Fed. Cir. 1984); In re Baird, 29 USPQ 2d 1550 (CAFC 1994) and In re Fritch, 23 USPQ 2nd. 1780 (Fed. Cir. 1992)). In re Gorman, 933 F.2d 982, 987, 18 USPQ2d 1885, 1888 (Fed. Cir. 1991) (in a determination under 35 U.S.C. § 103 it is impermissible to simply engage in a hindsight reconstruction of the claimed invention; the references themselves

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must provide some teaching whereby the applicant's combination would have been obvious); In re Dow Chemical Co., 837 F.2d 469,473, 5 USPQ2d 1529, 1531 (Fed. Cir. 1988) (under 35 U.S.C. § 103, both the suggestion and the expectation of success must be founded in the prior art, not in the applicant's disclosure). The applicants disagree with the Examiner why one skilled in the art with the knowledge of the references would selectively modify the references in order to arrive at the applicants' claimed invention.

Obviousness cannot be established by combining the teachings of the prior art to produce the claimed invention absent some teaching, suggestion, or incentive supporting this combination, although it may have been obvious to try various combinations of teachings of the prior art references to achieve the applicant's claimed invention, such evidence does not establish *prima facie* case of obviousness (In re Geiger, 2 USPQ 2d. 1276 (Fed. Cir. 1987)). There would be no reason for one skilled in the art to combine Auerbach taken with Paul in view of Chapman and Mück. For the above reasons, this rejection should be withdrawn.

In view of the above amendment, applicant believes the pending application is in condition for allowance.

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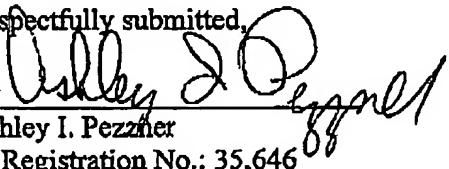
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A one month extension fee has been paid. Applicant believes no fee is due with this response. However, if a fee is due, please charge our Deposit Account No. 03-2775, under Order No. 05587-00327-US from which the undersigned is authorized to draw.

Respectfully submitted,

By 
Ashley I. Pezzner
Registration No.: 35,646
CONNOLLY BOVE LODGE & HUTZ LLP
1007 North Orange Street
P.O. Box 2207
Wilmington, Delaware 19899
(302) 658-9141
(302) 658-5614 (Fax)
Attorney for Applicant

ENCLOSURE: TERMINAL DISCLAIMER

STANDARD USED BY GENERAL MOTORS

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ENGINEERING
STANDARDS
EUROPE

**TEST METHOD
PRÜFVERFAHREN**

GME 60 271

Determination of the Release of Formaldehyde from Moulded Parts for the Vehicle Interior
Bestimmung der Formaldehydabgabe von Formteilen für den Fahrzeuginnenraum

Revisions/Änderungen

Date/Datum	Sym/Pos	Description/Beschreibung	Appd/Gen
08. 1.94		<p>Was Issue No. 1, Date of Issue 04/94 Titel changed References added Procedure with reference to VDA method 275 reworked War Ausfertigungs-Nr. 1, Datum 04/94 Titel geändert Referenzen zugefügt Durchführung in Anlehnung an VDA-Methode 275 überarbeitet</p> <p style="text-align: right;"><i>Joh</i></p>	
		<p>Fax Nr. 06 91 305 1709.1 DAL 8.2.96 An Hm. W. Grolik, Fa Herbst von A. OPEL AG, Zentrallabor</p> <p style="text-align: right;">Selten</p> <p><i>GME 60271.</i></p> <p>Telefax</p> <p><i>Fax Nr. 06 91 305 1709.1 DAL 8.2.96</i></p> <p><i>An Hm. W. Grolik, Fa Herbst</i></p> <p><i>von A. OPEL AG, Zentrallabor</i></p> <p><i>Selten</i></p> <p>Dieses Blatt unterliegt nicht dem Änderungsdienst</p> <p>No change service</p>	

Approved/Genehmigt

H. Schach

Adam Opel AG
TDC-Central Laboratory
TEG-Zentrallaboratorium

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GM	TEST METHOD PRÜFVERFAHREN	GME 60 271												
<p>Reference is made to the following national standards:</p> <table> <tr> <td>DIN EN 312</td> <td>08.93</td> <td>Wood-based panels; determination of moisture content</td> </tr> <tr> <td>VDA 275</td> <td>07.94</td> <td>Moulded composites and fleeces for vehicle; determination of formaldehyde release; test procedure called modified flask method</td> </tr> </table>			DIN EN 312	08.93	Wood-based panels; determination of moisture content	VDA 275	07.94	Moulded composites and fleeces for vehicle; determination of formaldehyde release; test procedure called modified flask method						
DIN EN 312	08.93	Wood-based panels; determination of moisture content												
VDA 275	07.94	Moulded composites and fleeces for vehicle; determination of formaldehyde release; test procedure called modified flask method												
<h3>1 Scope</h3> <p>This test method describes a measuring process for determining the release of formaldehyde from moulded parts for the vehicle interior. The test method was elaborated with reference to VDA 275.</p>														
<h3>2 Principle</h3> <p>For this test, test pieces of a particular mass and dimension are fastened above distilled or demineralized water in a sealed 1-l polyethylene bottle and stored at a constant temperature for a specified length of time. Afterward, the bottles are cooled and the formaldehyde absorbed in the distilled water is determined. The quantity of formaldehyde is ascertained in relation to the dry weight ($\mu\text{g/g}$) and surface ($\mu\text{g/cm}^2$) of the test piece.</p> <p>The formaldehyde is determined by photometric analysis using the acetylacetone method. In an aqueous solution, formaldehyde reacts with acetylacetone in the presence of ammonium ions to become 3,5-diacetyl-1,4-dihydroquinidine (DDQ). The resulting colour complex is yellow with maximum absorption at a wavelength of 412 nm.</p>														
<h3>3 Test Apparatus and Equipment</h3>														
<h4>3.1 Test Apparatus</h4>														
<p>3.1.1 Precision weighing device with scale gradation $\pm 1 \text{ mg}$.</p>														
<p>3.1.2 Well-ventilated heating cabinet which is capable of maintaining a temperature of $(103 \pm 2)^\circ\text{C}$ in accordance with DIN.</p>														
<p>3.1.3 Heating cabinet which is capable of maintaining a temperature of $(60 \pm 2)^\circ\text{C}$ in accordance with DIN.</p>														
<p>3.1.4 Spectrophotometer.</p>														
<p>3.1.5 1-l polyethylene bottle with hook implement integrated in the cover (see fig. 1).</p>														
<h4>3.2 Reagents</h4>														
<p>3.2.1 Acetylacetone p.a.</p>														
<p>3.2.2 Ammonium acetate p.a.</p>														
<p>3.2.3</p> <table> <tr> <td>Iodine solution c(I₂)</td> <td>= 0,05 mol/l</td> </tr> <tr> <td>Sodium thiosulphate c(Na₂S₂O₃)</td> <td>= 0,1 mol/l</td> </tr> <tr> <td>Caustic soda solution c(NaOH)</td> <td>= 1 mol/l</td> </tr> <tr> <td>Sulphuric acid c(H₂SO₄)</td> <td>= 1 mol/l</td> </tr> <tr> <td>Starch solution</td> <td>1 % m/m</td> </tr> <tr> <td>Formaldehyde solution p.a.</td> <td>37 %</td> </tr> </table>			Iodine solution c(I ₂)	= 0,05 mol/l	Sodium thiosulphate c(Na ₂ S ₂ O ₃)	= 0,1 mol/l	Caustic soda solution c(NaOH)	= 1 mol/l	Sulphuric acid c(H ₂ SO ₄)	= 1 mol/l	Starch solution	1 % m/m	Formaldehyde solution p.a.	37 %
Iodine solution c(I ₂)	= 0,05 mol/l													
Sodium thiosulphate c(Na ₂ S ₂ O ₃)	= 0,1 mol/l													
Caustic soda solution c(NaOH)	= 1 mol/l													
Sulphuric acid c(H ₂ SO ₄)	= 1 mol/l													
Starch solution	1 % m/m													
Formaldehyde solution p.a.	37 %													
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TEST METHOD
PRÜFVERFAHREN

GME 60 271

Folgende nationale Normen kommen in dieser Vorschrift zur Anwendung:

DIN EN 322	08.83	Holzwerkstoffe; Bestimmung des Feuchtegehaltes
VDA 275	07.84	Formteile für den Fahrzeuginnenraum; Bestimmung der Formaldehydabgabe; Maßverfahren nach der modifizierten Flaschen-Methode

1 Zweck

Diese Prüfverfahren beschreibt ein Maßverfahren zur Bestimmung der Formaldehydabgabe von Formteilen für den Fahrzeuginnenraum. Das Prüfverfahren wurde in Anlehnung an VDA 275 erstellt.

2 Prinzip

Bei der Prüfung werden Probekörper einer bestimmten Masse und Abmessung über destilliertem oder demineralisiertem Wasser in einer geschlossenen 1 l-Polyethylenflasche befestigt und bei konstanter Temperatur über eine definierte Zeit gelagert. Danach kühlst man die Flaschen ab und bestimmt im destillierten Wasser den absorbierten Formaldehyd. Die ermittelte Formaldehydmenge wird auf das trockene Proben Gewicht ($\mu\text{g/g}$) und auf die Probenoberfläche ($\mu\text{g/cm}^2$) bezogen. Die Formaldehydbestimmung erfolgt photometrisch nach der Acetylaceton-Methode. Formaldehyd reagiert in wässriger Lösung mit Acetylaceton in Gegenwart von Ammonium-Ionen zu 3,5-Diacetyl-1,4-Dihydrolukidin (DDL). Der entstehende Farokomplex besitzt eine gelbe Farbe mit einem Absorptionsmaximum bei einer Wellenlänge von 412 nm.

3 Prüfgeräte und Prüfmittel

3.1 Prüfgeräte

3.1.1

Präzisionswaage mit Skaleneinteilung $\pm 1 \text{ mg}$.

3.1.2

Gut bei offener Wärmeschrank, der in der Lage ist, eine Temperatur von $(103 \pm 2)^\circ\text{C}$ gemäß DIN zu halten.

3.1.3

Wärmeschrank, der in der Lage ist, eine Temperatur von $(80 \pm 2)^\circ\text{C}$ gemäß DIN zu halten.

3.1.4

Spektralphotometer.

3.1.5

1 l-Polyethylenflasche mit im Deckel integrierter Hängevorrichtung (siehe Abb. 1).

3.2 Prüfmittel

3.2.1

Acetylaceton p.a.

3.2.2

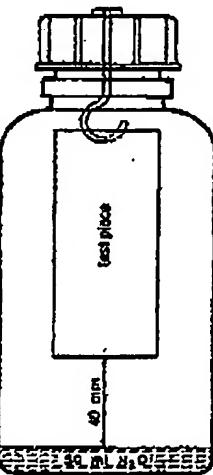
Ammoniumacetat p.a.

3.2.3

Iodösulig. c(I ₂)	= 0,05 mol/l
Natrium Iodosulfat c(Na ₂ S ₂ O ₃)	= 0,1 mol/l
Natronlauge c(NaOH)	= 1 mol/l
Schwefelsäure c(H ₂ SO ₄)	= 1 mol/l
Stärke Lösung	1% m/m
Formaldehydlösung p.a.	37%

Dok. Nr. 00000000000000000000000000000000

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GM	TEST METHOD PRÜFVERFAHREN	GME 60 271
<p>4 Test Pieces and Number of Test Pieces</p> <p>4.1 Taking of Test Pieces</p> <p>The test pieces shall be taken from suitable and representative points spread evenly across the width of the moulded part. They must be taken 50 mm from the edge of the moulded part.</p> <p>4.2</p> <p>The size of each test piece equals 40 mm x 100 mm x thickness. If samples of these dimensions cannot be taken due to the size of the moulded part, or if the distance of 50 mm to the edge of the part cannot be kept, this test may also be performed using smaller test pieces. Here, care must be taken to ensure that all the edges of test pieces are freshly cut. At least 4 samples must be taken from each moulded part, whereby 3 parts are used for determining formaldehyde and one part is used to determine the moisture content.</p> <p>4.3</p> <p>If testing of the test piece cannot take place immediately, they must be stored in cool, air-tight conditions with a minimal air volume. Storage time must not exceed 5 days.</p> <p>5 Procedure</p> <p>5.1 Determining the Moisture Content</p> <p>The moisture content is determined in accordance with DIN EN 322.</p> <p>5.2 Determining the Release of Formaldehyde</p> <p>5.2.1</p> <p>Before the analysis commences, the test pieces are balanced on the analyzing scales to exactly 0,01 g. The length, width and thickness of the sample, used to calculate its surface area, must be determined precisely to 0,1 cm.</p> <p>To fasten the test piece, a hole corresponding to the diameter of the hook is made in its centre, 10 mm from the upper edge.</p> <p>50 ml of distilled or demineralized water are pipetted into each of the 1-l polyethylene bottles.</p> <p>Once the test piece has been attached to the hook, the bottle is sealed and stored for 3 hours in a heating cabinet at +60°C.</p> <p>When the test time is up, the containers are removed from the heating cabinet. After being left to stand at room temperature for 60 minutes, the samples are taken out of the bottles.</p> <p>For each series of measurements, a dummy test piece and a calibration standard are to be handled in the same manner as the other samples.</p>  <p>Fig. 1: Structure of test apparatus</p>		

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GM

TEST METHOD
PRÜFVERFAHREN

GME 60 271

4 Probekörper und Anzahl der Probekörper

4.1

Die Probekörper sind gleichmäßig verteilt über die Breite an geeigneten und repräsentativen Stellen des Formteils zu entnehmen. Die Probenahme muß 50 mm vom Formteilrand erfolgen.

4.2

Die Größe des Prüfkörpers beträgt jeweils 40 mm x 100 mm x Dicke. Kann aufgrund der Größe des Formteils kein Prüfkörper der angegebenen Maße entnommen werden oder kann der Abstand von 50 mm zum Rand des Formteils nicht eingehalten werden, so kann die Messung auch mit Probekörpern kleineren Ausmaßes durchgeführt werden. Hierbei ist darauf zu achten, daß alle Kanten des Prüfkörpers frisch angeschnitten sind.

Pro Formteil werden mindestens 4 Probekörper entnommen, davon 3 Teile für die Formaldehydbestimmung und ein Teil für die Feuchtebestimmung.

4.3

Kann mit den entnommenen Probekörpern die Prüfung nicht sofort erfolgen, so sind diese luftdicht, kühl und mit mindestens Luftvolumen zu lagern. Die Lagerzeit darf 5 Tage nicht überschreiten.

5 Durchführung

5.1 Bestimmung des Feuchtegehaltes

Der Feuchtegehalt wird nach DIN EN 322 bestimmt.

5.2 Bestimmung der Formaldehydabgabe

5.2.1

Die Probekörper werden vor Beginn der Analyse auf der Analysenwaage auf 0,01 g genau ausgewogen. Die Länge, Breite und Dicke des Probekörpers, die zur Berechnung der Oberfläche herangezogen werden, sind auf 0,1 mm genau zu bestimmen.

Zur Belebung werden die Probekörper 10 mm vom oberen Rand mittig mit einer in Abhängigkeit zum Haken stehenden Bohrung versehen.

In die 1 l-Polyethylenflasche wird jeweils 50 ml destilliertes oder demineralisiertes Wasser pipettiert.

Nach Anbringen des Probekörpers am Haken wird das Gefäß verschlossen und über 3 Stunden im Wärmeschrank bei +60°C verwahrt.

Nach Ablauf der Prüfzeit werden die Gefäße aus dem Wärmeschrank genommen. Nach 60 Minuten Standzeit bei Raumtemperatur werden die Prüfkörper aus den Gefäßen entfernt.

Je eine Blindprobe und ein Standard der Kalibrierung sind für jede Meßreihe in gleicher Weise wie die Proben zu behandeln.

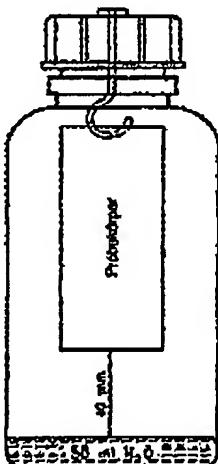


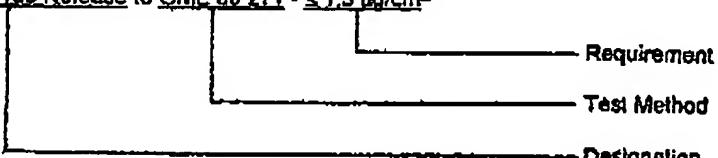
Abb. 1: Aufbau der Prüfapparatur

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<p>5.2.2 Photometry</p> <p>5.2.2.1 Reagents</p> <p>5.2.2.1.1 Acetylacetone solution</p> <p>1 ml of acetylacetone is pipetted into a 250-ml metering flask, filled up to 250 ml with demineralized water and shaken hard.</p> <p>5.2.2.1.2 Ammonium acetate solution</p> <p>50 g of ammonium acetate are measured out in a 250-ml metering flask, dissolved in demineralized water and topped up to 250 ml.</p> <p>5.2.2.2 Determining the concentration of formaldehyde in the aqueous solution</p> <p>10 ml of the aqueous solution resulting from the process described in 5.2.1 is transferred to a wide-necked, 50-ml Erlenmeyer flask with a ground surface, along with 10 ml of acetylacetone solution and 10 ml of ammonium acetate solution. The wide-necked Erlenmeyer flask is sealed, shaken and heated in a +40 °C water bath for 15 minutes.</p> <p>After a cooling period of one hour away from the light, the test piece is measured at 412 nm against demineralized water using the spectrophotometer.</p> <p>5.2.2.3 Calibration</p> <p>The calibration curve is produced by means of a formaldehyde standard solution, the formaldehyde concentration of which is determined by iodometric titration. From this standard solution, a calibration solution with a mass concentration of 15 µg formaldehyde/ml is formed. The formaldehyde concentration of the standards should lie between 0 and 15 µg/ml. The gradient of the calibration curve is determined mathematically by means of linear regression. To ascertain the calibration curve, the gradient and the determined coefficient of correlation are given.</p>									
<h2>6 Evaluation</h2> <h3>6.1 Moisture Content</h3> <p>The moisture content H of the moulded part in % is derived from the following formula:</p> $H = (m_1/m_0 - 1) \cdot 100 \%$ <p>Here: m_1 is the mass of the test piece in grammes before drying m_0 is the mass of the test piece in grammes after drying</p> <h3>6.2 Calculating the Release of Formaldehyde</h3> <p>6.2.1 In 1 g/g:</p> $\frac{(A_S - A_B) \cdot f - V \cdot (100 + H)}{m_1 \cdot 1000} \cdot F = \mu\text{g HCHO/g abs. dry sample}$ <p>6.2.2 In Relation to the Surface Area of the Test Piece (O_p) in $\mu\text{g/cm}^2$:</p> $(A_S - A_B) \cdot f - V / O_p = \mu\text{g HCHO/cm}^2 \text{ surface of test piece}$ <p>A_S Extinction of analyzed test piece A_B Extinction of dummy value f Gradient of calibration function in µg/ml m₁ Mass of the test sample in g before drying H Moisture content of the test material in percent V Volume of the absorber solution (50 ml) F Factor for calculating the result of the analysis in g (µg/g); F = 10 O_p Surface of test piece in cm² Abs.dry Absolutely dry test material</p>									
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<p>5.2.2 Photometrie</p> <p>5.2.2.1 Reagenzien</p> <p>5.2.2.1.1 Acetylacetonlösung</p> <p>1 ml Acetylaceton werden in einen 250 ml-Meßkolben pipettiert, mit demineralisiertem Wasser auf 250 ml aufgefüllt und kräftig geschüttelt.</p> <p>5.2.2.1.2 Ammoniumacetatlösung</p> <p>50 g Ammoniumacetat werden in einem 250 ml - Meßkolben eingewogen, in demineralisiertem Wasser gelöst und auf 250 ml aufgefüllt.</p> <p>5.2.2.2 Bestimmung der Formaldehydkonzentration in der wässrigen Lösung</p> <p>10 ml der unter 5.2.1 erhaltenen wässrigen Lösung werden in einem 50 ml Weithalsseriemeyerkolben mit Schliff mit 10 ml der Acetylacetonlösung und 10 ml der Ammoniumacetatlösung versetzt. Der Weithalsseriemeyerkolben wird verschlossen, geschüttelt und für 15 Minuten bei +40 °C im Wasserbad erwärmt.</p> <p>Nach einerständiger Abkühlung unter Lichtverschluß wird die Probe bei 412 nm gegen demineralisiertes Wasser im Spektralphotometer gemessen.</p> <p>5.2.2.3 Kalibrierung</p> <p>Die Kalibrierkurve wird mittels einer Formaldehyd-Standardlösung erstellt, deren Formaldehydkonzentration durch Iodometrische Titration bestimmt wird. Von dieser Standardlösung wird eine Kalibrierlösung mit der Masse konzentration 15 µg Formaldehyd/ml erstellt.</p> <p>Die Standards sollen in ihren Formaldehydkonzentrationen zwischen 0 und 15 µg/ml liegen.</p> <p>Die Steigung der Kalibrierkurve wird über lineare Regression rechnerisch bestimmt. Zur Beurteilung der Kalibrierkurve wird die Steigung und der ermittelte Korrelationskoeffizient angegeben.</p>									
<p>6 Auswertung</p> <p>6.1 Feuchtegehalt</p> <p>Der Feuchtegehalt H des Formteils in % ergibt sich aus der folgenden Formel:</p> $H = (m_1/m_0 - 1) \cdot 100 \%$ <p>Dabei ist: m_1 die Masse des Prüfkörpers vor dem Trocknen in Gramm m_0 die Masse des Prüfkörpers nach dem Trocknen in Gramm</p> <p>6.2 Berechnung der Formaldehydabgabe</p> <p>6.2.1 in µg/g:</p> $\frac{(A_S - A_B) \cdot f \cdot V \cdot (100 + H)}{m_1 \cdot 1000} \cdot F = \mu\text{g HCHO/g} \text{ atro Probe}$ <p>6.2.2 bezogen auf die Oberfläche des Prüfkörpers (O_p) in µg/cm²:</p> $(A_S - A_B) \cdot f \cdot V / O_p = \mu\text{g HCHO/cm}^2 \text{ Oberfläche der Probe}$ <p>A_S Extinktion der analysierten Probe A_B Extinktion des Blindwertes f Steigung der Kalibrierfunktion in µg/ml m₁ die Masse des Prüfkörpers vor dem Trocknen in g H Feuchtegehalt des Probematerials in Prozent V Volumen der Absorbenzlösung (50 ml) F Faktor zur Berechnung des Analysenergebnisses in g (µg/g); F = 10 O_p Oberfläche des Prüfkörpers in cm² atro absolut trockenes Probenmaterial</p>									
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<p>7 Expression of Results The formaldehyde concentration shall be expressed as the average value of three determination in µg/g and µg/cm², and referred to this test method.</p> <p>8 Coding System The formaldehyde concentration shall be specified on drawings and in material specifications as follows:</p> <p>e.g.: <u>Formaldehyde Release to GME 60 271 - ≤ 1.5 µg/cm²</u></p>  <p>9 Deviations Deviations from the conditions of this test method (e.g. storage, temperature, time etc.) shall have been agreed upon. This shall be specified on part drawings, material specifications etc. and shall be indicated in all test certificates, reports etc.</p>									
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TECHNISCHE
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TEST METHOD PRÜFVERFAHREN

GME 60 271

7 Angabe des Ergebnisses

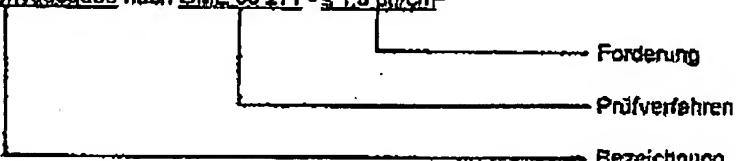
Die Formaldehydkonzentration wird unter Hinweis auf dieses Prüfverfahren als arithmetischer Mittelwert aus drei Bestimmungen in $\mu\text{g/g}$ und $\mu\text{g}/\text{cm}^2$ angegeben.

8 Angabe in technischen Dokumenten

In Zeichnungen, Werkstoffvorschriften und dergl. wird die Formaldehydkonzentration wie folgt angegeben:

z.B.:

Formaldehydabgabe nach GME 60 271 - ≤ 1,5 $\mu\text{g}/\text{cm}^2$



9 Abweichungen

Abweichungen von den Bedingungen dieses Prüfverfahrens (z.B. Lagertemperatur, Zeit usw.) müssen vereinbart werden. Diese Bedingungen sind in Teilezeichnungen, Werkstoffvorschriften und dergl. vorgeschrieben und müssen in Prüfzeugnissen, Berichten und dergl. angegeben werden.